

Technical Memorandum 33-650

*Long-Time Dynamic Compatibility of
Elastomeric Materials With
Hydrazine*

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**JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY
PASADENA, CALIFORNIA**

September 1, 1973

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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PREFACE

This work was performed by the Propulsion Division of the Jet Propulsion Laboratory for the George C. Marshall Space Flight Center of the National Aeronautics and Space Administration. The work was administered under the technical direction of the Materials Division of the MSFC Astronautics Laboratory with Mr. John T. Schell acting as technical representative.

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ABSTRACT

The tensile property surfaces for two elastomeric materials, EPT-10 and AF-E-332, have been generated in air and in liquid hydrazine environments using constant strain rate tensile tests over a range of temperatures and elongation rates. These results have been used to predict the time-to-rupture for these materials in hydrazine as a function of temperature and amount of strain covering a span of operating times from less than a minute to twenty years. The results of limited sheet-folding tests and their relationship to the tensile failure boundary are presented and discussed.

I. INTRODUCTION

The objective of this program was to establish a basis for predicting the long-term property characteristics of elastomeric materials when used in hydrazine propellant systems. The application of particular interest was propellant tank bladders or diaphragms for space shuttle vehicle auxiliary propulsion systems. These systems must be capable of ten-year/100-mission operation lifetimes with minimum refurbishment.

Two candidate elastomeric diaphragm materials, EPT-10 and AF-E-332, were selected for evaluation of their elastomer "tensile property surface" in air and in hydrazine. Using the tensile property surface for the elastomer and defining its failure boundary in terms of stress, strain, time and temperature, it should be possible to predict the ten-year failure characteristics, based on data obtained over a period of a few weeks.

Although considerable property data are available on these two materials, their applicability has been focused mainly on one- or two-year lifetimes and less than 50 flexing cycles. During this program, mechanical property data were obtained in air and in hydrazine to allow the prediction of long-time (ten-year) fatigue, creep, relaxation, tensile strength, elastic modulus and elongation characteristics of EPT-10 and AF-E-332 in air and in liquid hydrazine at temperatures between 275 and 383 K (2 to 110°C). From the "property surface" data obtained in hydrazine and in air plus related stress relaxation data obtained in nitrogen at temperatures to 458 K (185°C), it was concluded that the elastomer property surface in nitrogen would be identical to that in air in the temperature range of interest (275 to 383 K).

The experimental technique used to develop the "property surface" for the two elastomers in hydrazine is described in detail in Appendix A. This appendix was prepared in the form of an ASTM paper for presentation at the ASTM 1973 Annual Meeting in Philadelphia, Pa, June 24-29, 1973.

Limited testing was conducted also on the effects of severe folding of the elastomer sheets and the effect of temperature on elastomer swelling in hydrazine.

A start was made on the systematic compilation of all available properties of these materials in air and hydrazine, as referred to in the Appendix C of Ref. 1, but it was soon evident that the range of test conditions and material process parameters were so diverse that the task could not be completed within the program scope. Also, new data were being continuously generated by the material vendors and users. It is recommended that anyone who is interested contact JPL and the vendors directly for specific properties of interest until a systematic compilation of properties can be prepared.

II. DISCUSSION OF RESULTS

A. Strain-to-Failure

The major applicable results of these measurements for predicting elastomer lifetimes are summarized in Figs. 1 and 2, which present the tensile strain versus time-to-rupture at various temperatures in hydrazine for EPT-10 and AF-E-332. These curves are based on the strain-to-failure data obtained with ring specimens at constant strain rates in air and hydrazine shown correlated in Fig. A-5 of Appendix A. The experimental data showed that for both materials the effects of time and temperature on the shape of the strain-to-failure curves were, surprisingly, the same in air and in hydrazine. Thus, the time-to-failure, at a given strain level, would be the same in hydrazine as in air. In using these curves for predicting failure in hydrazine, the loading due to constant tensile strain, or constant load or cyclic fatigue may be traced out as strain versus time on Figs. 1 and 2 to intercept the predicted cumulative time-to-failure curves at the given operating temperature.

The effect of temperature on material life (i.e., time-temperature shift factor) was found to be the same for both the EPT-10 and AF-E-332.

The value of the time-temperature shift factor, $\log a_T$, which was the basis for the horizontal location of the curves in Figs. 1 and 2 was checked by several independent methods. These shift factors were first calculated as described in Appendix A by correlating the stress-to-failure and the strain-to-failure data with strain rate and temperature. Also, five specimens of AF-E-332 were subjected to stress relaxation tests at different temperatures in hydrazine at which time load versus time was measured at a fixed strain of 100%. In addition, the plots of the stress-strain curves (low strain modulus) versus temperature and strain rates were used to calculate the values of $\log a_T$. These $\log a_T$ values were plotted and found to correlate well with the Williams, Landel, and Ferry (WLF) equation of Refs. 2 and 3:

$$\log a_T = \frac{-8.86 (T - T_0)}{101.6 + (T - T_0)}$$

where T_0 was selected as 305 K to provide the best data correlation (Fig. 3).

Comparing the properties of the two materials EPT-10 and AF-E-332, which are both silica-filled ethylene propylene terpolymers, shows them to have appreciable differences in mechanical properties (strength and modulus) and also different swelling responses to hydrazine (Fig. 4). Nevertheless, in the effect of temperature on time-to-rupture, they were essentially equal to each other and to related filled and unfilled organic polymers such as SBR gum vulcanizates (Ref. 2).

It may be useful in emphasizing the sensitivity of these types of elastomers to temperature, to note that the time-to-rupture at the 293 K (20°C) level (based on $\log a_T$ values) decreases by a factor of almost ten for every 8 K (8°C) increase in temperature (Fig. 3). Between 293 K (20°C) and 373 K (100°C) the time-to-rupture for both materials would decrease by a factor of 10^5 ; or, at a particular strain level, from 20 years to two hours.

Also, it may be observed from the rupture data scatter that a factor of 100 in time-to-rupture uncertainty may exist due to the fact that failures are initiated at microscopic surface flaws and propagate in the filled elastomer, somewhat in the manner of brittle materials.

Comparing the lifetimes of EPT-10 and AF-E-332 at 100% strain at 20°C (Figs. 1 and 2), it would appear that the EPT-10 would last longer at the same strain than AF-E-332 although both would last in excess of one year. However, these curves also reflect the fact that the EPT-10 was a softer material and, therefore, under less stress at 100% strain. This may indeed contribute to longer life in flexing application. However, in severe folding and in cases where the materials are held folded (or creased) for long periods, the effects of compression set on the inside of the fold may be of major importance in determining the failure mode. Apparently these two materials do differ in this property as described in the following paragraphs. Also, greater tear resistance is claimed for the AF-E-332; a property which would have to be established by an appropriate test method.

B. Folding Failures

For the more typical mode of biaxial loading, such as flexing or severe folding, one might conservatively estimate that if the failure life predicted for the corresponding temperature and strain level in uniaxial loading is insufficient, the biaxially strained life would also be insufficient. To evaluate the validity of such an approach, limited folding tests were conducted.

The maximum strain level measured in a single sharply folded elastomer sheet is less than 100% at the surface of the fold. If this sheet is folded again, creating a double fold, as in Fig. 4, the maximum surface strain measured was approximately 100% in each direction at the nose of the double fold.

Double-folded specimens of the EPT-10 and AF-E-332 were prepared and held at 100°C in water and also in air at 100 and 130°C. No failures occurred during test times that exceeded those predicted for failure at 100% uniaxial strain.

Although the biaxial surface strain of 100% measured at the nose of a double folded sheet would seem more severe than 100% uniaxial strain, apparently the sharp negative gradient of local strain normal to the material surface at this location was effective in retarding the initiation or propagation of a failure crack or tear. For instance, razor cuts intentionally inflicted at the nose of the folds did not self-propagate.

However, when these double-folded specimens which had been held at elevated temperatures for more than 48 h were unfolded, it was apparent that the EPT-10 had experienced more permanent compression set at the inside crease than the AF-E-332. When these same specimens were then double folded in the reverse direction and heated to 130°C, the EPT-10 failed in less than 5 min at the nose of the fold. In the AF-E-332 only a small crack developed away from the fold, but no failure was induced at the nose of the fold.

A satisfactory correlation between these folding failures and uniaxial properties has not been developed. However, all experience with these elastomers would indicate that the relative effects of temperature and time on failure would remain the same for both materials stressed in air or in hydrazine.

C. Swelling in Hydrazine

Swelling of EPT-10 and AF-E-332 in hydrazine was measured as a function of time at three temperatures: 278, 298, and 343 K (5, 25, and 70°C). The results are presented in Fig. 5. The specimens used were the tensile test rings cut from the 1.96-mm (0.077 in) thick sheets described in Appendix A. From these results it appears that the materials swell faster at the higher temperature (343 K) but do not reach the same weight increase as obtained at room temperature and below. This may have been due in part to desorption and evaporation of hydrazine during specimen cooling between removal of specimen from the hot hydrazine and the time of weighing. The specimens were weighed within 30 to 40 sec of removal from the hydrazine after excess liquid was blotted with a paper tissue.

Rates of weight increase are a function of both specimen size and geometry. The large difference in level of weight increase between the EPT-10 and AF-E-332 is due to either the character of the silica filler used or the higher crosslinking. A weight increase of over 30% for EPT-10 was recorded, compared to 3% for the AF-E-332.

III. CONCLUSIONS

(1) The "tensile property surfaces" for EPT-10 and AF-E-332 elastomers in liquid hydrazine have been defined, and the failure boundaries as a function of time and temperature are predicted for times exceeding 20 years, based on the experimental data obtained and assuming no degradation.

(2) The temperature dependence of the mechanical properties of EPT-10 and AF-E-332, both in air and in hydrazine follow the same relationship derived for a wide range of filled and unfilled elastomers.

(3) Limited testing of folded sheets of EPT-10 and AF-E-332 indicate the potential applicability of both these materials at prescribed temperature limits over a ten-year period; however, additional testing is recommended to establish the relationship between the tensile failure boundary and failures due to folding and flexing typical of the intended applications.

(4) Although the results of this program do not clearly establish the superiority of either EPT-10 or AF-E-332 as a diaphragm material, they will provide a basis for selecting future test methods and evaluating test data for applicability to the long-time multicycle application of interest.

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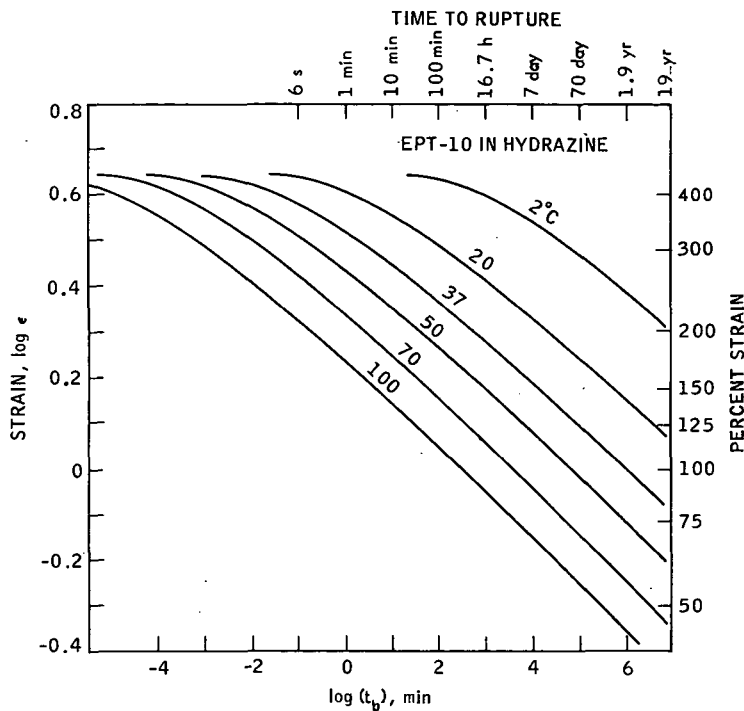


Fig. 1. Tensile strain versus time to rupture in hydrazine as a function of temperature for EPT-10 elastomer

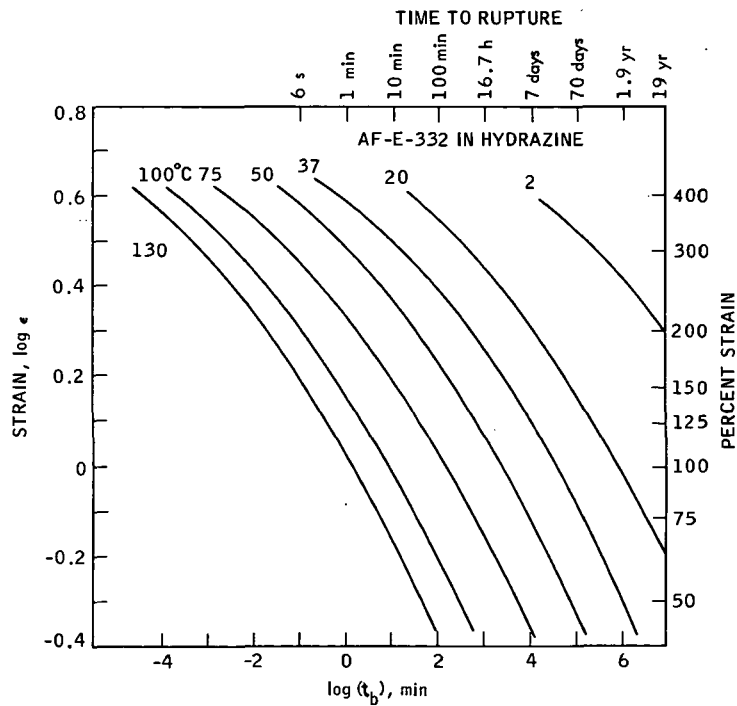


Fig. 2. Tensile strain versus time to rupture in hydrazine as a function of temperature for AF-E-332 elastomer

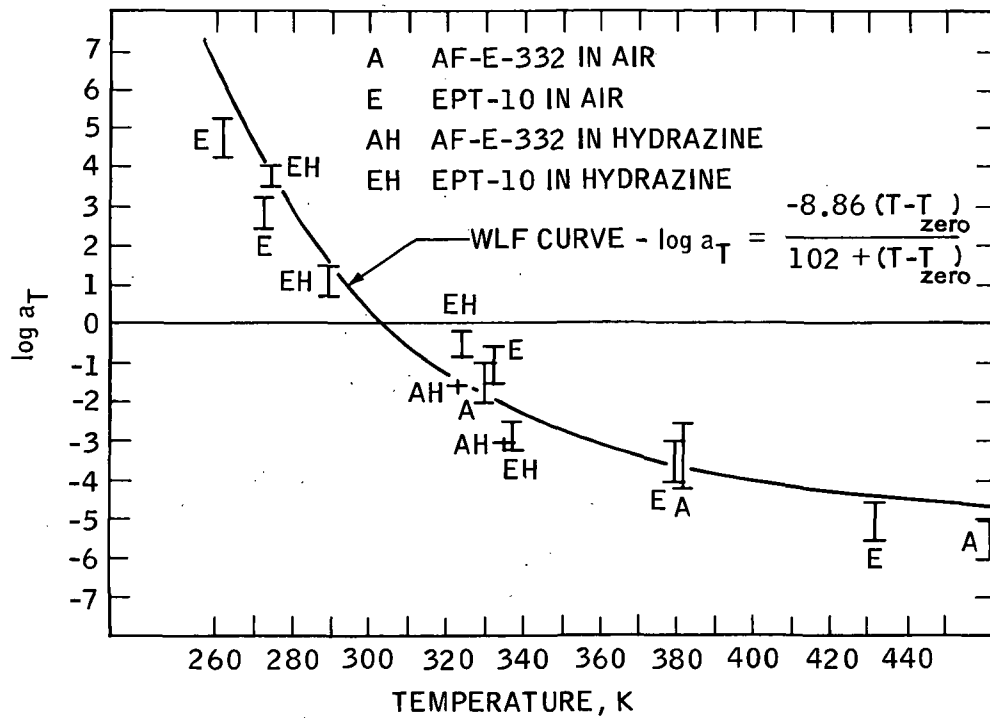


Fig. 3. Experimental values of $\log a_T$ in air and hydrazine compared with WLF Equation (Ref. 2)

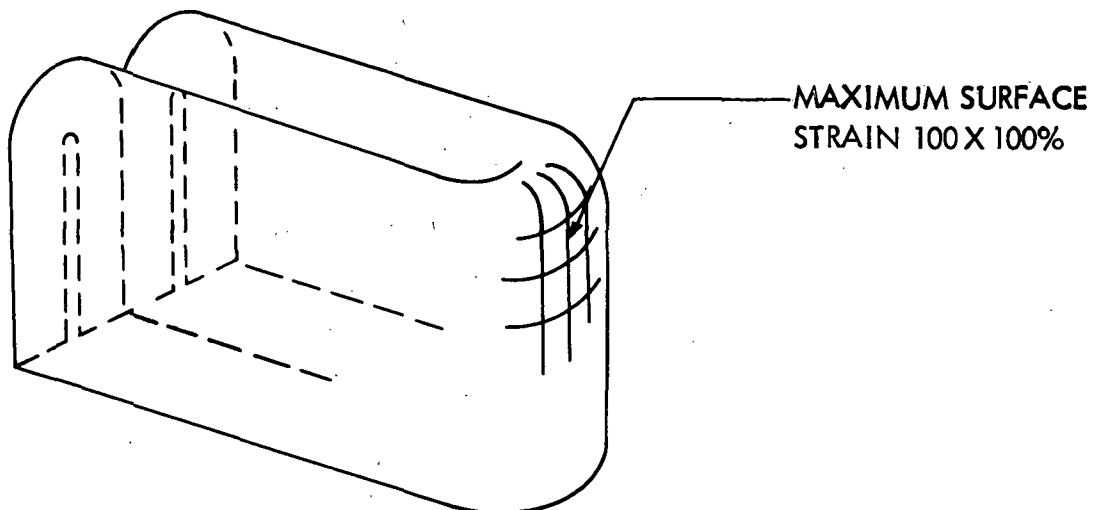


Fig. 4. Double-folded elastomer sheet

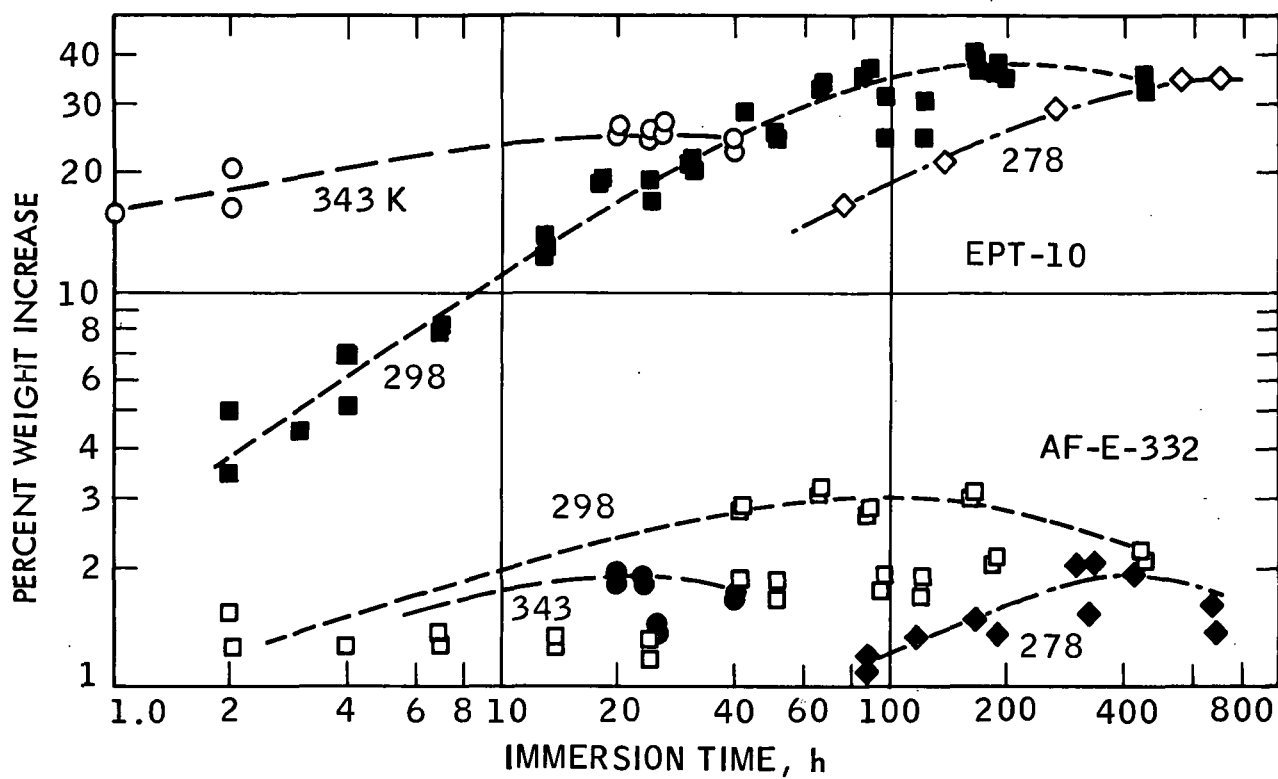


Fig. 5. Swelling of elastomers EPT-10 and AF-E-332

APPENDIX A

EXPERIMENTAL PROCEDURES OF PREDICTING LONG-TIME DYNAMIC COMPATIBILITY OF TWO ETHYLENE PROPYLENE ELASTOMERS WITH HYDRAZINE

I. INTRODUCTION

Elastomeric materials in contact with liquid hydrazine may experience changes in physical properties as manifested by swelling, reduction in tensile strength, and possible leaching of constituents (Refs. 1 and 2). When these elastomers are employed as components subject to cyclic loading such as propellant tank bladders or diaphragms, it is important also to be able to predict their long-time survivability under dynamic stressing conditions in hydrazine.

This paper describes a technique for characterizing elastomeric materials in liquid hydrazine during short-time tests to provide a basis for predicting the useful life of bladders or diaphragms subject to multiple propellant expulsion cycles over long time periods.

The method is applied to those candidate elastomers which have survived a preliminary compatibility examination, typically static immersion at various temperatures in hydrazine with inspection for visible evidence of incompatibilities, and measurements of any swelling or softening of the elastomer (Ref. 3).

In normal practice, the elastomers surviving the compatibility screening are removed from the hydrazine and tested for changes in tensile strength, modulus, elongation-at-break, etc. However, this approach only evaluates changes from immersion, if any, by comparison of properties before and after, but does not provide information on the level of elastomeric properties while in the hydrazine environment, nor give information which directly allows for a prediction of dynamic life time.

II. METHOD FOR PREDICTING LONG-TIME BEHAVIOR

The approach selected as a basis for predicting long-time behavior was based on the existence and approximate invariance of the tensile

property surface for elastomers. The concept of the property surface is based on the fact that the mechanical response of an elastomer can be considered in terms of the three variables: stress σ , strain ϵ , and time t . Hence, a convenient representation is an analytic surface in stress, strain, and time, space. The effect of temperature is incorporated into the time scale by the relationship $\log t/a_T$, where the value of $\log a_T$ is the experimentally determined time-temperature shift factor.

The development of a tensile property surface which relates these parameters by a single analytic surface with a single rupture failure boundary is described by R. F. Landel and R. F. Fedors in Ref. 4. A typical tensile property surface for Viton B rubber in air taken from the reference is shown in Fig. A-1.

The surface is conveniently generated by measuring the uniaxial stress-strain response of the given elastomer as a function of strain rate and temperature. Typically, it is necessary to test the elastomer at about 10 different strain rates at each of 10 to 15 test temperatures. Although this represents a considerable expenditure of time and effort, knowledge of the surface is of fundamental importance in understanding and predicting the mechanical response of an elastomer.

The response of an elastomer to any uniaxial input will simply be a path traced out on the surface. For example, the path traced out during a creep experiment (i.e., constant load) starting at point A in Fig. A-1 and terminating at the point B, will be the curve AB, which is generated by the intersection of this surface with a plane parallel to the strain-time plane and which passes through both A and B. The projection of this path to the strain-time plane is depicted by the lower curve AB; this latter curve corresponds to creep data as normally plotted. Similar consideration can be used for experiments carried out under other test modes. Thus, the path traced out on the surface by a stress relaxation (i.e., fixed strain) experiment carried out by starting from the same point A used for the creep experiment, is shown in Fig. A-1 as the curve AC.

Rupture on this surface is represented graphically as a discontinuity or boundary which can be considered to result from the intersection of the physical property surface with another surface representing failure. In Fig. A-1 this rupture condition is represented by the terminating line on

the right. Hence, the specimen undergoing creep and following the surface along path AB will break when this path intersects the boundary (at point C). From the general nature of the surface, it is evident that starting from the same initial point A, the creep path will reach the boundary before the stress-relaxation path and, hence, the lifetime under creep conditions will be less than that under stress relaxation conditions.

It has also been found experimentally (Ref. 4) that fatigue life in uniaxial tensile tests bears the same relationship to the property surface as the other modes of testing. In Fig. A-1 a fatigue trace would have a saw-tooth waveform that moves across the surface until it reaches the failure boundary. The implication of this criterion is that fatigue is less dependent on the number of cycles than on the total time under load. A current experimental program under way at JPL is evaluating the fatigue life of elastomers over a wide range of load cycling frequencies.

The effect of changes in the crosslink density of the elastomer on the location of the property surface has also been studied for three chemically different types of rubber, viz., a hydrocarbon, a fluorocarbon, and a fluorosilicone rubber. For all three types, the effect of a change in crosslink density is simply to change the time scale of the experiment analogously to the change in time scale observed when the temperature is changed. For example, if the crosslink density were inadvertently increased by 20%, the lifetime under stress would decrease by a factor of four. Such changes in crosslink density could occur due to variations in material formulation and processing conditions.

In many applications, the elastomer is subjected to biaxial deformations (e.g., flexing and folding) while the experience presented above was limited to tests carried out in uniaxial deformation. This, again, is an area where more investigation can profitably be carried out. However, it can be said that the failure time in biaxial deformation can never exceed that under conditions of uniaxial deformation. Hence, the results obtained with the uniaxial test can be used to assess the promise of a candidate material. For example, if the candidate material does not have sufficient lifetime in a uniaxial test, it should not be a candidate for an application involving biaxial deformation.

With test data obtained over a sufficient range of temperatures and strain rates, the time dependencies of both the stress and strain-at-break may be readily determined for up to 13 decades of time (i.e., times from less than 1 sec to more than 20 years). The test temperatures must be above the glass transition temperature but below the temperature at which chemical degradation would be rapid compared to the time scale of the experiment.

III. EXPERIMENTAL PROGRAM

A. Approach

The objective of this program was to determine the property surface in hydrazine for two candidate elastomers in order to assess and rank their potential for a long-term dynamic application. The selected elastomers were two ethylene propylene terpolymers designated EPT-10 and AF-E-332. The ability to develop a complete property surface in hydrazine was limited by the limited range of experimental test temperatures available for conducting the tensile tests in liquid hydrazine, which were temperatures between the freezing point 274.5 K and the boiling point 386.5 K. For reasons of safety and limited test experience, the actual test temperatures used were limited to values between 275 and 341 K.

The experimental approach was to establish the property surface for each material first in air over a larger temperature range (233 to 453 K) to provide a basis for evaluating the influence of hydrazine immersion and, if possible, a basis for extrapolating the hydrazine property surface data. Thus, rupture data obtained in less than two hours per specimen at a maximum of 341 K in hydrazine would be used in determining the time dependence of rupture for ten years of operation at temperatures between 275 and 384 K in hydrazine.

IV. SPECIMEN PREPARATION

A single batch of each elastomer to be evaluated was ordered from the manufacturer in the form of $152 \times 152 \times 1.96$ mm sheets of a sufficient quantity to complete all the contemplated tensile testing and other related

tests such as swelling, stress relaxation, and folding tests. The ethylene propylene terpolymer EPT-10 was purchased from Pressure Systems, Inc., of Los Angeles, Ca. The AF-E-332 was an EPT compound to which silica, Teflon and HYSTL Resin B-3000 had been added to optimize its properties. This material was purchased from TRW Systems of Redondo Beach, Ca.

The test specimen rings were cut from the sheets using a rotating two-bladed cutter that cut the inside and outside circumferences simultaneously. The test specimen ring size was 19.2 mm ID by 22.3 mm OD. After cutting, the rings were individually measured, weighed, and bagged for subsequent testing. Approximately 300 rings of each material were thus prepared.

V. TENSION TESTS IN AIR

Tension tests in air were conducted in a controlled temperature enclosure mounted in an Instron tensile test machine. Twelve test temperatures were selected between 233 and 453 K and controlled by circulating heated or cooled air through the enclosure. The crosshead speeds were set at ten values from 8.5 to 0.0085 mm/s. During testing the test rings were mounted over hooks formed from 4.7-mm diam steel rods. The recorded data consisted of load-time curves on a strip chart. The initial point on each load-time curve was determined by extending the initial straight portion of the curve backward to the zero load line. This correction was employed, because the rings started out circular and the force required to straighten the ring caused an indeterminant start point for the load-time curve. Since the elongations at rupture were in the 250 to 400% range, the effect of initial differences in ID and OD stresses were assumed to be negligible.

Selected points from the load-time curves were tabulated and used in subsequent computer data reduction calculations. A total of 120 rings were tested to rupture for each material.

VI. TENSION TESTS IN HYDRAZINE

Conducting a large number of tensile rupture tests in liquid hydrazine at elevated temperatures required special test planning considerations. Liquid hydrazine (N_2H_4) is a reactive compound used as a monopropellant and also as a bipropellant in combination with an oxidizer, such as nitrogen tetroxide. It is thermodynamically unstable and is very sensitive to trace amounts of catalyst, which cause it to exist in a state of continuous decomposition. This decomposition rate is a function of both temperature and the type or amount of catalyst which may be present. At ambient temperatures ~ 293 K and in the absence of a catalyst, however, the average decomposition rate of N_2H_4 is minimal. Although liquid hydrazine has been heated under pressure to temperatures above 533 K with very little decomposition, the hydrazine vapors can present a hazard. Air saturated with hydrazine at any temperature above 313 K is a flammable mixture. Therefore, the testing approach which was developed utilized a specially designed carousel, or turret, arrangement (Fig. A-2) which provided the capability for testing eight ring specimens in hydrazine as a single operation at one temperature with minimum handling and exposure of the hydrazine.

The design consisted of eight individual AISI 304 steel test chambers filled with hydrazine and contained inside a larger water-filled tank mounted in the Instron tensile test machine. In this test setup, ring specimens were mounted individually on AISI 304 steel hooks inside each of the eight test chambers. The shafts of the upper hooks extended through caps on the top of each chamber to permit attachment to the Instron loading grip as each specimen chamber was successively rotated into test position. The eight chambers were mutually interconnected by a common hydrazine feed manifold which had an external entrance and exit port. After the eight specimens were mounted in the chambers, the chambers were evacuated and hydrazine was drawn into the chambers, filling them all simultaneously to the same level from a common hydrazine supply. During testing, dry nitrogen gas was passed through the hydrazine manifold and the ullage space in each chamber. Temperature control was achieved by circulating water through the outer tank from a thermally regulated externally located separate water bath. Temperatures could thus be set at values between 275 and 368 K.

This test arrangement allowed the test chambers to be filled once each day with hydrazine and heated (or cooled) to a selected temperature. Then the eight specimens were tested in tension successively at eight selected crosshead speeds, from 8.5 to 0.085 mm/s. All the ring specimens tested in hydrazine were presoaked for a minimum of 48 h in hydrazine at room temperature in order to reach equilibrium swelling. Preceding swelling studies showed this to be a sufficient swelling time.

The recorded data was handled similarly to that for the air test data. The five selected temperatures were 275, 291, 311, 325 and 341 K. Thus, 40 ring specimens of each material were tested to rupture in hydrazine.

VII. DATA ANALYSIS

The load-time data from the Instron strip charts were tabulated for each ring and used as the input to a data analysis computer program for all subsequent calculations. From the computer program output, plots were prepared to display graphically on translucent graph paper the relationships of $\log (\sigma_b^{297/T})$ versus $\log t_b$ and $\log \epsilon_b$ versus $\log t_b$ for each specified temperature, where

σ_b = tensile stress at failure, N/m^2

T = test temperature, K

t_b = time to failure, min

ϵ_b = strain at failure

After preparing the individual plots for each test temperature, the series of plots for each material were placed on a light table on top of each other and shifted along the time ($\log t_b$) axis until a single (best fit) master curve was derived (Ref. 4).

As with similar data for other polymeric materials, it was found that a number of iterations were required to achieve a reasonable fit because of the considerable data scatter. To illustrate the principle involved, the two

plots in Fig. A-3 may be studied. The plots on the left represent the strains and times at failure for each temperature level. The lowest curve represents data taken at the highest temperature. All test times lie between ten seconds and one hundred minutes. In the figure at the right the data have been shifted horizontally with respect to the reference curve at 310 K to form a single curve. The amount of shift is $\log a_T$ so that the final curve is plotted as $\log \epsilon_b$ versus $\log (t_b/a_T)$. The final values of $\log a_T$ were obtained by shifting both the strain and stress data with the requirement that the relationship between $\log a_T$ and test temperature be a continuous smooth function of the form

$$\log a_T = \frac{-C_1(T - T_0)}{C_2 + (T - T_0)}$$

where

T_0 = the selected reference temperature, K

T = test temperature, K

C = experimentally determined constant for the material tested.

The actual data taken for EPT-10 in hydrazine showed less scatter than the data taken in air and less than that for the AF-E-332 material in either air or hydrazine. Figure A-3 presents the time dependence for the breaking strain for EPT-10 before and after shifting by the $\log a_T$ factor.

The final plots of the time dependence of the breaking stress and strain for both EPT-10 and AF-E-332 in air and in hydrazine are presented in Figs. A-4 and A-5. A single equation for the value of the temperature shift factor, $\log a_T$, found to be satisfactory for correlating both materials in air and in hydrazine was

$$\log a_T = \frac{-8.86(T - 305)}{102 + (T - 305)}$$

It is possible that slightly different constants might be established for each material if the data scatter could be reduced.

VIII. SUPPLEMENTARY TESTING

One of the more striking observations made from comparing the EPT-10 data taken in air with that in hydrazine was that, while there was a definite reduction in tensile strength and a concomitant increase in the breaking strain in hydrazine, for longer times and higher temperatures the strain data in hydrazine coincided with the air test data. The significance of this is that the long-time strain to failure for EPT-10 is the same in both air and hydrazine. These data, therefore, provided a potential basis for long time failure prediction. However, to assure the validity of these relationships, selected additional tests were designed to produce failures in somewhat longer test times to check the effect of temperature and low strains on the location of the lower end of the ϵ_b versus (t_b/a_T) curve.

Two types of supplementary tests were conducted. These tests were conducted in air, since the same ϵ_b versus (t_b/a_T) relationship was obtained in both air and hydrazine. Additional tests in hydrazine with both elastomers are planned as a further follow-on to this work which will provide additional confirmation and better definition of the failure curves.

In one test series performed in air, ring specimens of EPT-10 were stretched between a series of fixed rods mounted on an aluminum block in such a way that fixed strains of 100, 125, 150, 200, and 250% could be imposed on the rings. The rings were stretched between the rods, several at each strain value, and the whole assembly was placed in a preheated air oven at temperatures of 343, 373, or 403 K. The combination of strain and oven temperature was chosen so that the predicted failure times would range between one minute and a few hours. The failure times for such a test mode can be predicted directly from the plots of $\log \epsilon$ versus $\log (t_b/a_T)$. The data from these oven tests are indicated in Fig. A-5 by the flagged data points. Additional tests at lower temperatures and longer test times are also being carried out.

The second type of test conducted was to form double folds of each material from strips 25 by 75 mm and clamp them in the folded position with a steel spring binder clip. These were placed initially in boiling water for times up to 12 hours without a failure. Another set of double folds was placed in the oven at temperatures from 373 to 403 K for 48 hr without

failure. Although the maximum surface biaxial strain which occurred at the nose of the double fold was 100%, failures were never initiated at this location. Failures in these double-fold specimens for both the EPT-10 and AF-E-332 were induced finally by reversing the double folds after the 48-hr exposure and replacing them in the oven at 403 K.

Since these were supplementary and exploratory type tests, the test conditions were not precisely controlled. These initial results did confirm the fact that the severely folded elastomers would not fail in times shorter than those predicted by the uniaxial tensile failure data for 100% strain.

IX. DISCUSSION

A working graph derived from the failure data for EPT-10 in hydrazine showing the strain to failure as a function of time and temperature (in degrees Centigrade) is shown in Fig. A-6. In the use of such a working graph one must keep in mind the scatter in the experimental data (see Fig. A-5) which in some instances spanned two or three decades of time. However, the plot is very useful in demonstrating the sensitivity of the life of these materials to temperature and strain levels. For instance, elastomers which would fail in 16 hr at room temperature ($\sim 20^{\circ}\text{C}$) at 225% strain, would fail in 20 min at 50°C . Also, material dynamically stressed at room temperature in hydrazine to 100% strain or less, could last for more than ten years.

These results will be particularly useful in selecting test parameters and designing further experiments to be conducted in hydrazine, such as long-time creep or fatigue tests. For the application of these results to predicting the life of a propellant tank bladder or diaphragm for a long-life multicycle mission, an experiment should be designed to produce failure during cyclic folding in a controlled manner in hydrazine. The results of this present program would indicate that failures of EPT-10 could be produced in a few weeks if the tests were conducted at temperatures above 343 K.

In this paper, no attempt has been made to specifically compare the performance of EPT-10 with AF-E-332 because the purpose here was to develop and evaluate a test method. Any performance differences which are

apparent from these data should be confirmed by additional specific tests that have a more direct relationship to the intended use of the material.

X. CONCLUSION

This program has demonstrated that the typical elastomer tensile property surface does exist for the EPT-10 and AF-E-332 materials in hydrazine and a method for defining these characteristics has been outlined. Additional experimental work is required to refine the failure curves for long operating times at low-strain levels and to establish the relationship between folding failures and the experimental tensile strain failure limits.

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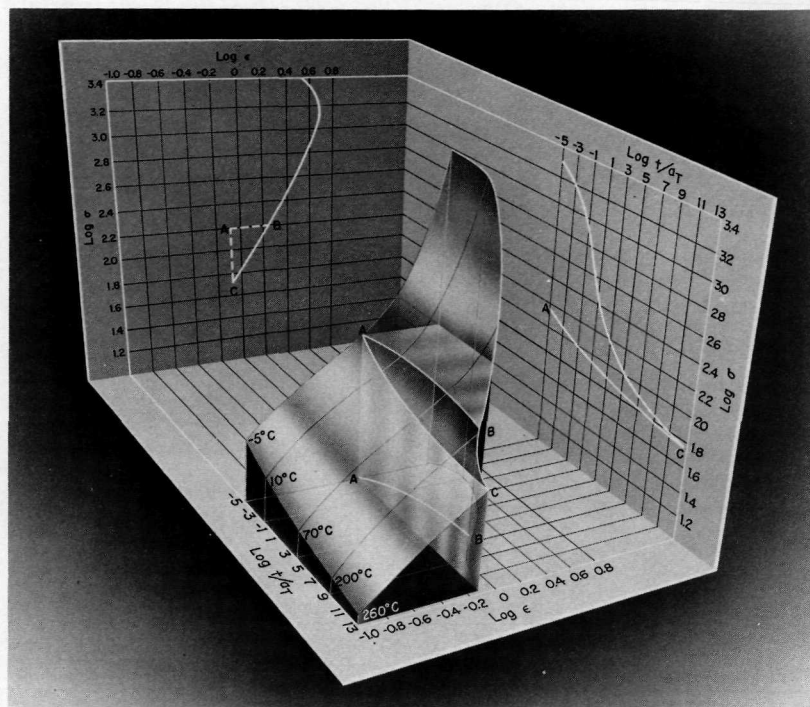


Fig. A-1. Tensile property surface for Viton rubber

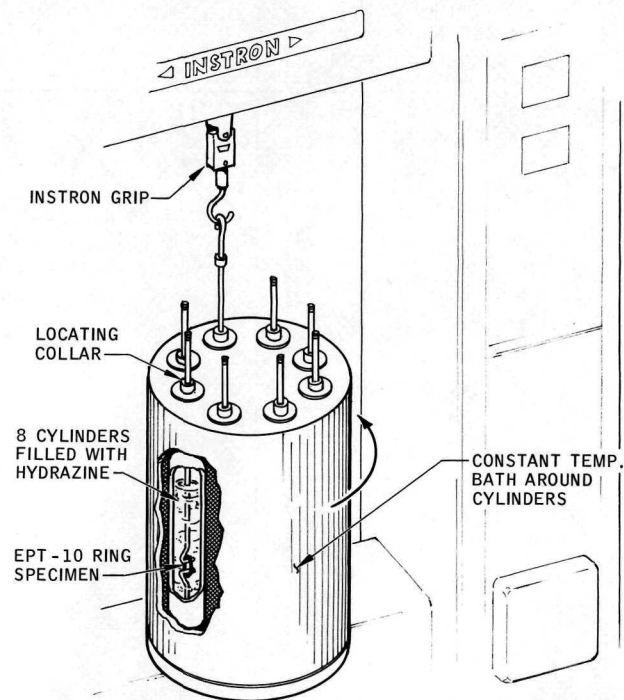


Fig. A-2. Turret action hydrazine ring test figure

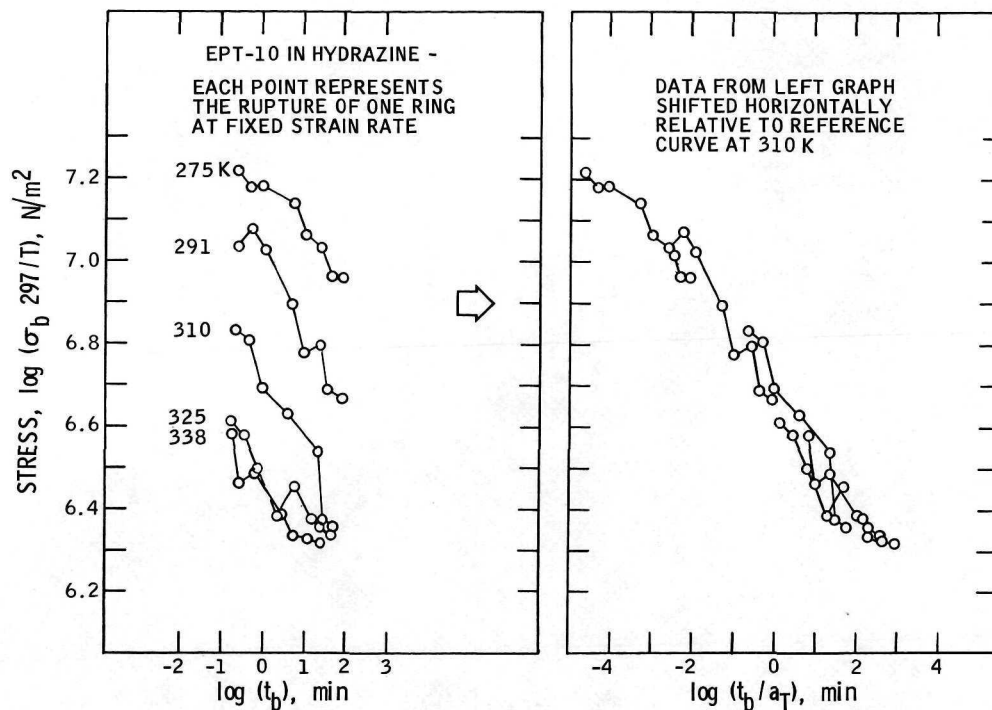


Fig. A-3. Illustration of the superposition of stress rupture data by shifting horizontally by factor $\log a_T$

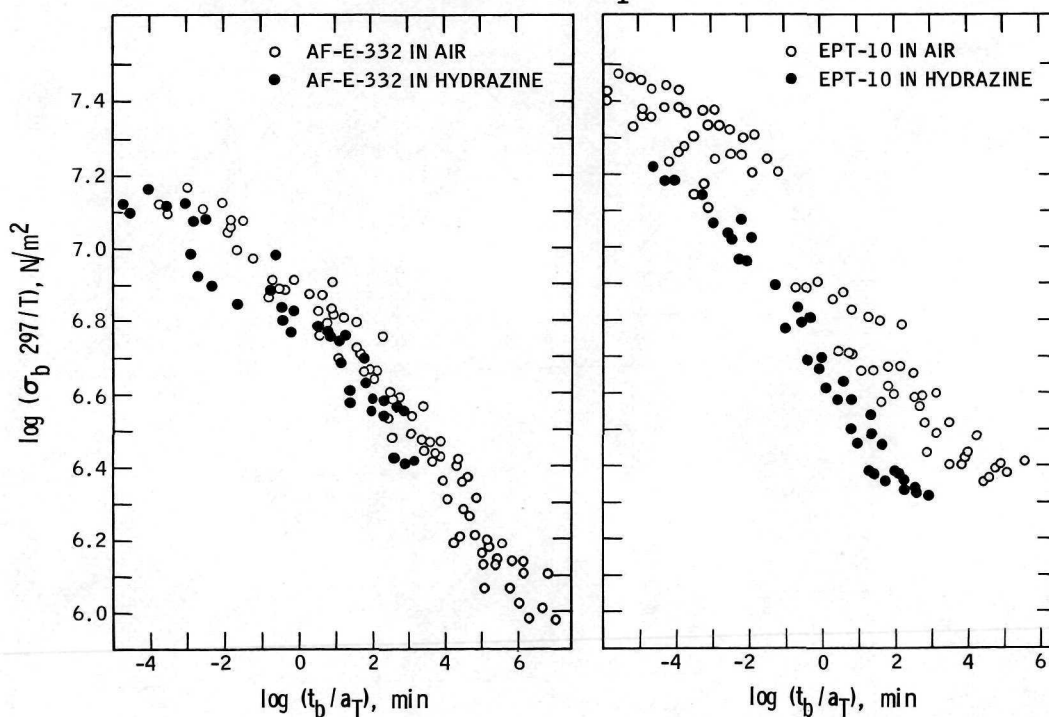


Fig. A-4. Tensile stress failure envelope for AF-E-332 and EPT-10 elastomers tested in air and in hydrazine at constant strain rates

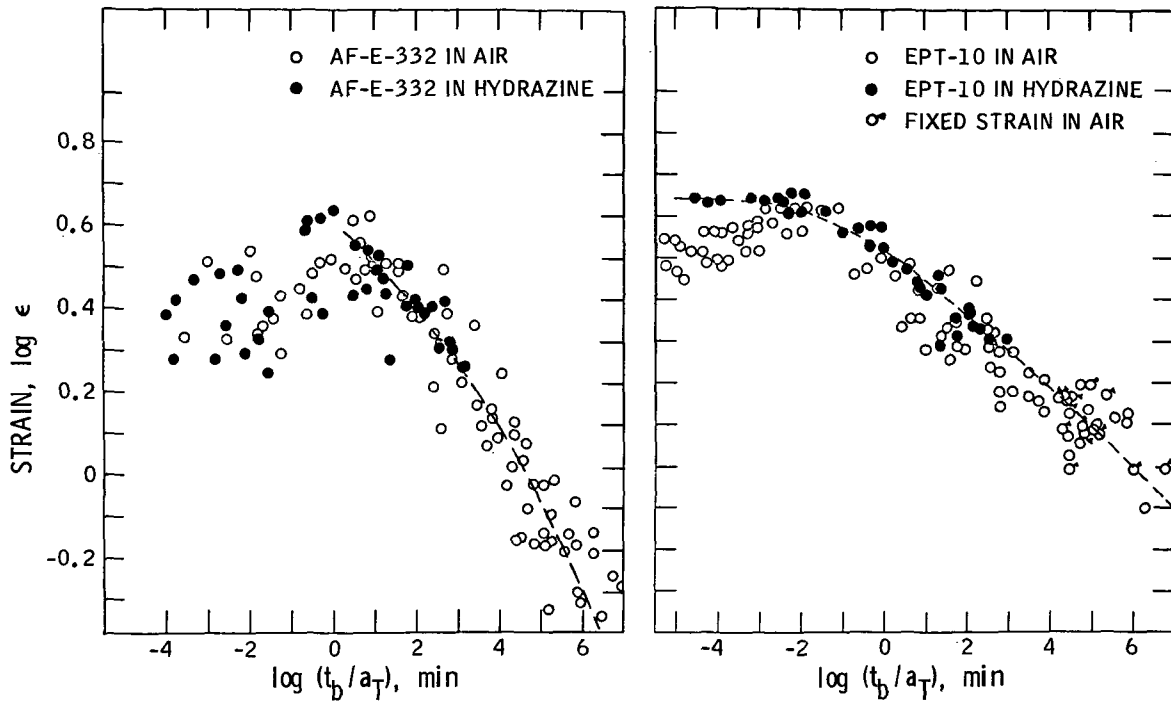


Fig. A-5. Tensile strain failure envelope for AF-E-332 and EPT-10 elastomers tested in air and in hydrazine at constant strain rates

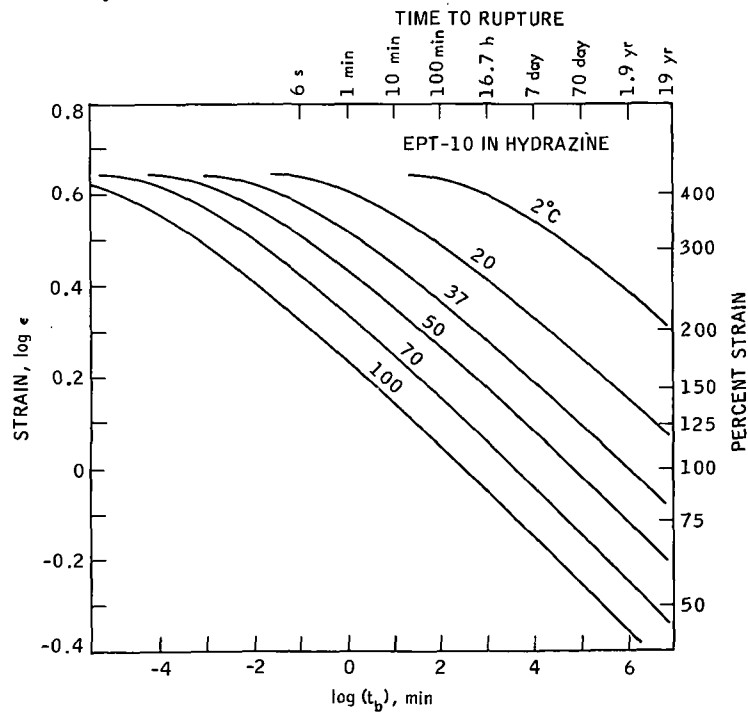


Fig. A-6. Tensile strain versus time to rupture in hydrazine as a function of temperature for EPT-10 elastomer